



ACQUISITION,
TECHNOLOGY
AND LOGISTICS

OFFICE OF THE UNDER SECRETARY OF DEFENSE

3000 DEFENSE PENTAGON
WASHINGTON, DC 20301-3000

June 15, 2001

MEMORANDUM FOR U.S. MISSION TO NATO, ARMAMENTS COOPERATION DIVISION
(ARMY ARMAMENTS OFFICER), PSC 81, APO AE 09724

SUBJECT: Draft STANAG 4543 (Edition 1) – “Explosives, Specification For Nto (3-Nitro-1,2,4-Triazol-5-One) For Deliveries from one NATO Nation to Another ”

Reference document, AC/310-D/185, 22 January 2001, SAB.

The U.S. Armed Forces ratifies the referenced agreement with comment.

Ratification and implementation details are as follows:

IMPLEMENTATION

	Forecast Date	Actual Date
<u>RATIFICATION REFERENCE</u>	<u>NAVY ARMY AIR FORCE</u>	<u>NAVY ARMY AIR FORCE</u>
Memo, OUSD(A&T) DATED AS ABOVE	8 June 2001	8 June 2001

NATIONAL IMPLEMENTING DOCUMENT: None, will use the STANAG

RESERVATIONS: None.

COMMENTS: See attached DA Form 4797-R.

The point of contact is Mr. James E. Elliott, DSN 880-3047, commercial (973) 724-3047.

Anthony J. Melita
U.S. Key Delegate
AC/310 Main Group

encl: as stated



cf:

Mr. Don Porada, Naval Ordnance Safety & Security Activity, Code N6, 23 Strauss Avenue, Bldg D-323, Indian Head, MD 20640-5555

Mr. William Collins, Air Armament Center, AAC/SES, 1001 North 2nd Street, Suite 366, Eglin AFB, FL 32542-6838

Mr. James Elliott, U.S. Army Armament Research, Development & Engineering Center (ARDEC), AMSTA-AR-QAW-S, Picatinny Arsenal, NJ 07806-5000

Dr. Ruth Doherty, Naval Surface Warfare Center, Indian Head Division, Code 920T, 101 Strauss Ave, Indian Head, MD 20640-5035

Mr. Chris Janow, U.S. Army Armament Research, Development & Engineering Center (ARDEC), AMSTA-AR-CCZ, Picatinny Arsenal, NJ 07806-5000

Mr. Stephen N. Tanner, Naval Air Warfare Center, Code 476400D, China Lake, CA 93555-6001

Mr. Sami Hoxha, U.S. Army Armament Research, Development & Engineering Center (ARDEC), AMSTA-AR-QAW-S, Picatinny Arsenal, NJ 07806-5000

Mr. Homesh Lalbahadur, U.S. Army Armament Research, Development & Engineering Center (ARDEC), AMSTA-AR-CCF-D, Picatinny Arsenal, NJ 07806-5000

Mr. Herbert Egbert, U.S. Army Developmental Test Command, CSTE-DTC-TT-M, 314 Longs Corner Road, Aberdeen Proving Ground, MD 21005-5055

Mr. Brent Knoblett, DOD Explosives Safety Board, Room 856C, Hoffman Bldg I, 2461 Eisenhower Ave, Alexandria, VA 22331-0600

Dr. Jerry Ward, DOD Explosives Safety Board, Room 856C, Hoffman Bldg I, 2461 Eisenhower Ave, Alexandria, VA 22331-0600

Doctrine Division (C426), Marine Corps Combat Development Center, 3300 Russell Road, Suit 318A, Quantico, VA 22134-5021

HQUSAF/SAF/IAQ, 1500 Wilson Blvd, 9th Floor, Arlington, VA. 22209

Mr. R. Sladden, Armaments CO-Operation Section, Defence Support Division, NATO Headquarters, Avenue Leopold III, 1110 Brussels, Belgium

4543 (E1) - Comments

NO. (a)	NATION (b)	PAGE (c)	PARA (d)	LINE (e)	COMMENT(S) (f)	REASON(S) (g)
1	US	1	2.1	3	Add "and melt-castable" between "explosive)" and "compositions."	NTO is used in AFX-645, a melt-cast TNT-based IHE explosive designed for use in the MK82 bomb.
2	US	2	TABLE 1	Particle Size Distribution	REQUIREMENTS: For melt-castable compositions no more than 20 percent of the NTO shall pass through a 212 micron (70 mesh) screen.	Detrimental to the processing operation with melt-castable formulations.
3	US	2	TABLE 1	ADD: Packing Density	A packing (bulk) density for the as-received particulate NTO shall be > 0.97 g/cc	This requirement insures that the particulate NTO will not contain a significant portion of cylindrical particles (log-like). Bulk densities lower than 0.97 g/cc are detrimental to processing.
4	US	2	TABLE 1	ADD: Test Method 12	Packing density is determined by mildly vibrating a known mass of particulate NTO to a final measurable volume.	Mass divided by the final measured volume equals the bulk or packing density of the particulate NTO.
5	US	7	11	ADD: 11.1. Thermogravimetric Analysis (TGA).	Thermogravimetric analysis may be used as an alternate method for determining volatile content.	This technique is a quantitative method for determining weight loss that can be attributed to volatile content at the 103 °C upper temperature.

NO (a)	NATION (b)	PAGE (c)	PARA (d)	LINE (e)	COMMENT (S) (f)	REASON (S) (g)
6	U.S.	7	11		The Determination of Volatile Substances (para 11) will only be meaningful if the wet NTO was dried (para4) at a comparatively low temperature, such as 45 degrees C. Any heating prior to execution of para 11 will drive off some volatile materials that were present in the original NTO. The temperature at which the NTO is dried should be reported.	Technical concerns
7	U.S.	7	12	Heading	Change heading of para 12 to "Particle Size/Particle Size Distribution"	To improve clarity

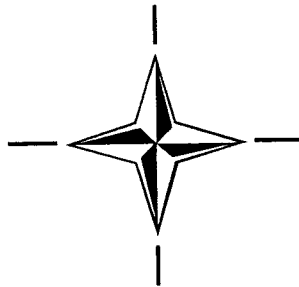
REVERSE OF DA FORM 4797-R, DEC 88

Encl 1

NATO/PfP UNCLASSIFIED

STANAG 4543
(Edition 1)

**NORTH ATLANTIC TREATY ORGANIZATION
(NATO)**




**NATO STANDARDIZATION AGENCY
(NSA)**

**STANDARDIZATION AGREEMENT
(STANAG)**

SUBJECT: EXPLOSIVES, SPECIFICATION FOR NTO (3-NITRO-1,2,4-TRIAZOL-5-ONE) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Promulgated on 26 August 2002


Jan H ERIKSEN
Rear Admiral, NONA
Director, NSA

NATO/PfP UNCLASSIFIED

STANAG 4543

(Edition 1)

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTESAGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Director, NSA under the authority vested in him by the NATO Military Committee.
2. No departure may be made from the agreement without consultation with the tasking authority. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.
3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

DEFINITIONS

4. Ratification is "In NATO Standardization, the fulfilment by which a member nation formally accepts, with or without reservation, the content of a Standardization Agreement" (AAP-6).
5. Implementation is "In NATO Standardization, the fulfilment by a member nation of its obligations as specified in a Standardization Agreement" (AAP-6).
6. Reservation is "In NATO Standardization, the stated qualification by a member nation that describes the part of a Standardization Agreement that it will not implement or will implement only with limitations" (AAP-6).

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

7. Page (iii) gives the details of ratification and implementation of this agreement. If no details are shown it signifies that the nation has not yet notified the tasking authority of its intentions. Page (iv) (and subsequent) gives details of reservations and proprietary rights that have been stated.

FEEDBACK

8. Any comments concerning this publication should be directed to NATO/NSA - Bvd Leopold III, 1110 Brussels - BE.

NAVY/ARMY/AIR

NATO STANDARDIZATION AGREEMENT
(STANAG)EXPLOSIVES, SPECIFICATION FOR NTO (3-NITRO-1,2,4-TRIAZOL-5-ONE) FOR DELIVERIES
FROM ONE NATO NATION TO ANOTHER

Related Documents: None

AIM

1. The aim of this agreement is to ensure that 3-nitro-1,2,4-triazol-5-one (NTO) shall possess properties which make it suitable for military use and to provide, within NATO, an acceptable basis for the procurement and certification of NTO.

AGREEMENT

2. Participating nations agree that NTO, proposed for military use, shall meet all the physical and chemical requirements of Table I of this document. The test procedures used to verify the requirements of Table I are described in Part III (paragraphs 6 through 14, inclusive) and the rejection criteria shall be in accordance with Part II, paragraph 5 of this document.

2.1 Use. NTO is intended for use in explosive systems, including General Purpose Bombs and missile systems. Typically, NTO is used as a component of main charge explosives such as PBX (plastic bonded explosive) compositions.

PART I - PHYSICAL AND CHEMICAL PROPERTIES

3. Physical and Chemical Properties. The requirements for the physical and chemical properties for NTO shall be as specified in Table I. Testing Methods are given as Part III of this document.

3.1 Structural Formula. The structural formula of NTO is given in figure 1 below.

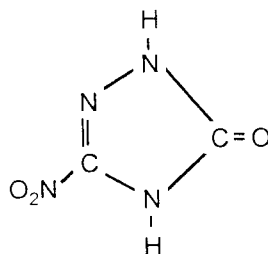


FIGURE 1. NTO STRUCTURAL FORMULA

TABLE I. CHEMICAL AND PHYSICAL PROPERTIES

PROPERTY	REQUIREMENTS	TEST METHOD Paragraph
Appearance	White to off-white crystal	
Decomposition Temperature by DSC		
3°C/min	271±1°C	7
5°C/min	277±1°C	
10°C/min	281±1°C	
NTO Purity	(1) > 99% NTO by HPLC or (2) > 99% NTO, by potentiometric method	8.2 8.3
Chlorine (as Chloride)	< 0.02%	9
Nitric Acid	< 0.05%	1
Volatile Substances	< 0.1%	11
Particle Size Distribution	Manufacturer will provide data and documentation of method used.	

PART II – SAMPLING AND REJECTION

4. **Sampling.** A lot of NTO shall consist of the quantity of material produced in a single batch; or when manufactured by a continuous process, a lot shall consist of the total quantity offered for acceptance at one time. A minimum of two (2) representative samples of at least 200 g each shall be taken from each container (e.g., drum or bag) of NTO by a sampling procedure which has been agreed upon by the purchasing authority. It will be necessary to dry samples before testing if the NTO was shipped wet.

5. **Rejection Criteria.** Failure of either of the samples will result in rejection of a container of NTO. If more than 30% of the samples from any given lot do not satisfy the requirements of this document, the entire lot will be rejected.

PART III - TESTING METHODS

6. **Warning.** Adequate safety precautions shall be taken during the processing, testing, and handling of the NTO Explosive to protect personnel from accidents, fires, or explosions, and to limit damage to equipment and processing areas. NTO Explosive required for testing shall be considered expendable. Disposal of the test samples after completion of tests shall be left to the discretion of the individual test authority.

7. Determination of Decomposition Temperature.

- 7.1 Definition. The temperature at which the solid material chemically breaks down into simpler compounds at atmospheric pressure as determined by differential scanning calorimetry (DSC).
- 7.2 Sample Preparation. A sample weight of between 0.5 to 1.0 milligram shall be used. The sample shall be prepared in accordance with the procedures detailed in the instruction manual of the specific DSC instrument used.
- 7.3 Test Procedure and Results. The sample shall be run in a nitrogen atmosphere in a sealed pan vented with a pin hole. The start temperature will be 50°C and the stop temperature will be 300°C with a heating rate no greater than 10°C per minute. In Table 1, temperatures have been provided for heating rates of 3, 5, and 10°C per minute. The operation, analysis, and calculational procedures of the DSC instrument used shall be followed for maximum accuracy and precise measurement of the DSC curve. The peak exotherm temperature shall be reported as the decomposition temperature.

8. Determination of NTO Purity.

- 8.1 Definition. NTO purity shall be measured by either HPLC analysis or a potentiometric technique.
- 8.2 HPLC Analysis.
- a. Sample Preparation for HPLC Analysis.
- (1) Standard sample - Dissolve 1.0 gram of pure NTO in water in a 100 ml volumetric flask. [The purity of the NTO standard should be $\geq 99\%$, as determined by either the potentiometric technique (paragraph 8.3) or by using a photodiode array detector to verify the UV spectrum (i.e., purity) of NTO.] Dilute to mark with HPLC grade water. Pipette 5.0 ml of this solution into a 10 ml volumetric flask. Dilute to mark with HPLC grade water.
 - (2) Sample solution - Accurately weigh 0.25 gram of the NTO sample into a 50 ml volumetric flask. Dilute to mark with HPLC grade water.
- b. Equipment for HPLC Analysis. A high performance liquid chromatograph (HPLC) system can be used to determine the weight percent of NTO in the sample.
- c. Materials and HPLC Conditions.
- The following operating conditions are given for information only.
- Mobile phase/eluent - 100 percent HPLC grade water plus IPCA (ion pairing agent); also, various proportions of acetonitrile and water could be used
 - Flow rate - 1.0 to 2.0 ml/minute
 - UV wave length - 214 nm
 - reverse phase column or cartridge C-18
 - run time - 15 minutes
 - sample size - 1.0 to 5.0 microliters

STANAG 4543

(Edition 1)

- d. Calculations. Calculate the concentration of NTO from its peak area, using the peak of pure NTO for comparison. The operation, analysis, and calculational procedures of the HPLC equipment used shall be followed for maximum accuracy and precise area measurements of the HPLC trace.

8.3 Potentiometric Method.

- a. Equipment. An automatic potentiometric titrator assembly comprised of a recording potentiometer fitted with an automatic burette and equipped with a 20 ml syringe and a combination glass-Ag/AgCl electrode can be used.
- b. Reagents. The reagents for this test method are:
- Tetrabutylammonium hydroxide (TBAH) 0.1 N (This is a commercial solution in methanol or in an isopropanol/methanol mixture. This solution must be stored between 16 and 24°C.).
 - Isopropanol/distilled water solution (97/3 percent ratio by volume).
 - Benzoic acid, analytical grade, dried at 65.5°C.
- c. Procedure.

The temperature of the TBAH solution should not vary by more than $\pm 1^\circ\text{C}$ during the determination of the TBAH titer and the NTO purity determination.

(1) Determination of TBAH Titer.

Transfer a weighed portion of approximately 0.1 gram of benzoic acid (weighed to ± 0.1 mg) to a 150 ml beaker. Add 100 ml of the aqueous isopropanol solution. Stir until completely dissolved. Then, using the potentiometric titrator, titrate with the TBAH 0.1 N solution. A minimum of three tests with benzoic acid and three tests without benzoic acid (blank tests) shall be conducted. The titrator determines the equivalence point, which is the point where the addition of a minimum amount of TBAH titration solution leads to a maximum change in potential.

The normality of TBAH is calculated as follows:

$$\text{Normality of TBAH} = \frac{(1000) (m)}{(122.1) (V_1 - V_o)}$$

Where: m: Weight of benzoic acid used (grams)

V_1 : Volume in ml of TBAH solution used to reach the equivalence point in the benzoic acid test. The average of three tests is used.

V_o : Volume in ml of TBAH solution used to reach the equivalence point for the blank test. The average of three tests is used.

122.1: Molecular weight of TBAH.

(2) Determination of NTO Purity.

Transfer a weighed portion of approximately 0.1 gram of NTO (weighed to ± 0.1 mg) to a 150 ml beaker. Add 100 ml of aqueous isopropanol. Stir until the NTO is completely dissolved. Then, using the titrator, titrate with the 0.1 N TBAH solution. A minimum of three tests shall be conducted. The titrator determines the equivalence point, which is the point where the addition of a minimum amount of TBAH titration solution leads to a maximum change in potential. The NTO purity is calculated as follows:

$$\text{NTO purity} = \frac{(130) (V_1) (N) (100)}{(m) (1000)}$$

Where: m: Weight of NTO (grams)

V_1 : Volume of TBAH solution used to reach the equivalence point. The average of three tests is used.

N: Normality of TBAH solution (obtained above)

130: Molecular weight of NTO

9. Determination of Chlorine Content (as Chloride)

- a. Equipment. An automatic potentiometric titrator assembly comprised of a recording potentiometer fitted with an automatic burette and equipped with a 5 ml syringe and the following electrodes:

- Ag as working electrode.
- Ag/AgCl in KCl/ K_2SO_4 as reference electrode.

- b. Reagents. The reagents for this test method are:

- 0.02 N $AgNO_3$ aqueous solution.
- 0.3 g/l NaCl aqueous solution.
- 30% concentrated HNO_3 Nitric Acid solution (1:1 ratio of concentrated nitric acid to water).
- distilled water.

- c. Procedure: Transfer a weighed portion of 5 ± 1.0 gram of NTO (weighed to ± 0.1 mg) to a 150 ml beaker. Add 100 ml of distilled water and stir until the NTO is completely dissolved. Add precisely 5 ml of the 0.3 g/l NaCl solution with a pipette. Add 5 ml of the 30% concentrated HNO_3 solution. Then, using the potentiometric titrator, titrate with the 0.02 N $AgNO_3$ solution. The titrator determines the equivalence point, which is the point where the addition of a minimum amount of $AgNO_3$ titration solution leads to a maximum change in potential. A minimum of three tests shall be conducted. A minimum of three tests without NTO shall also be conducted. Then Chloride Concentration is calculated as follows:

$$\% \text{ Cl} = \frac{35.5 \times (V_1 - V_0) (100) (0.02)}{(m) (1000)}$$

Where: m: weight of NTO (grams)

STANAG 4543
(Edition 1)

- V_1 : volume in ml of AgNO_3 solution used to reach the equivalence point with weight "m" of NTO. The average of a minimum of three tests is used.
- V_0 : Volume in ml of AgNO_3 solution used to reach the equivalence point without NTO. The average of a minimum of three tests is used.
- 0.02: Normality of AgNO_3 solution
- 35.5: Atomic weight of Chlorine

10. Determination of Nitric Acid Concentration

- a. Equipment: An automatic potentiometric titrator assembly comprised of a recording potentiometer fitted with an automatic burette and equipped with a 5 ml syringe and a combination glass-Ag/AgCl electrode can be used.
- b. Reagents: The reagents for this test method are:
- Tetrabutylammonium hydroxide (TBAH) 0.1 N (This is a commercial solution in methanol or in an isopropanol/methanol mixture. This solution must be stored between 16 and 24°C.).
 - Isopropanol/distilled water solution (97/3 percent ratio by volume).
 - Nitric Acid 0.1 N in aqueous solution.
- c. Procedure: Transfer a weighed portion of 5-10 gram of NTO (weighed to ± 0.1 mg) to a 150 ml beaker. Add precisely 1 ml of the 0.1 N nitric acid solution with a pipette. Add 100 ml of aqueous isopropanol. Stir until the NTO is completely dissolved. Then, using the titrator, titrate with the 0.1 N TBAH solution. The titrator determines the equivalence point, which is the point where the addition of a minimum amount of TBAH titration solution leads to a maximum change in potential. A minimum of three tests shall be conducted. A minimum of three tests without NTO shall also be conducted. The Nitric Acid Concentration is calculated as follows:

$$\% \text{HNO}_3 = \frac{(63) (V_1 - V_0) (100) (0.1)}{(m) (1000)}$$

Where: m: Weight of NTO used (grams)

- V_1 : Volume in ml of TBAH solution used to reach the equivalence point with "m" weight of NTO. The average of three tests is used.
- V_0 : Volume in ml of TBAH solution used to reach the equivalence point for the blank test. The average of three tests is used.
- 0.1: Normality of TBAH solution
- 63: Molecular weight of HNO_3

11. Determination of Volatile Substances. A 5-gram sample of NTO shall be placed in a 100 ml beaker and weighed. The beaker and contents shall be transferred to an air oven and heated at $103 \pm 2^\circ\text{C}$ for 2 hours. The beaker shall then be removed from the oven, placed in a desiccator until cool, and weighed. The volatiles content obtained by the difference in weight before and after heating shall be reported on a percentage basis.

$$\text{Weight percent volatiles} = \frac{(W_0 - W_1) (100)}{m}$$

Where: m : Weight of NTO used (grams)
 W_0 : Weight of beaker plus NTO initially (grams)
 W_1 : Weight of beaker plus NTO after drying (grams)

12. Note: Since there is significant difference between manufacturers for the particle size and particle size distribution of NTO, the manufacturer will provide particle size distribution data and documentation of the test method used when NTO is purchased.

PART IV - GENERAL INFORMATION

13. NTO is usually prepared by nitrating Triazolone (TO).

IMPLEMENTATION OF THIS AGREEMENT

14. This STANAG is considered implemented when a nation has issued the necessary orders and instructions putting the contents of this agreement into effect.

RATIFICATION AND IMPLEMENTATION DETAILS
STADE DE RATIFICATION ET DE MISE EN APPLICATION

EDITION: 1

N A T I O N	NATIONAL RATIFICATION REFERENCE DE LA RATIFICATION NATIONALE	NATIONAL IMPLEMENTING DOCUMENT NATIONAL DE MISE EN APPLICATION	IMPLEMENTATION / MISE EN APPLICATION					
			INTENDED DATE OF IMPLEMENTATION/ DATE PREVUE POUR MISE EN APPLICATION			DATE IMPLEMENTATION WAS ACHIEVED/ DATE REELLE DE MISE EN APPLICATION		
			NAVY MER	ARMY TERRE	AIR	NAVY MER	ARMY TERRE	AIR
BE								
CA	2441-4543 (A/DAPM 4-3) of/du 26.11.01	STANAG	01.02	01.02	01.02			
CZ	6/2-49/2001-1419 of/du 17.10.01	STANAG		02	02			
DA	FKO MAI2 204.69-S443 0101797-003 of/du 17.08.01	STANAG	01.03	01.03	01.03			
FR								
GE	BMVg-Fü S I 6 - Az 03-51-60 of/du 28.03.02	STANAG		11.02				
GR								
HU								
IT								
LU								
NL	2002001134 of/du 14.03.02	STANAG				07.02	07.02	07.02
NO								
PL								
PO								
SP								
TU								
UK	D/Dstan/12/15/4543 of/du 16.05.01	Not implementing/ Ne met pas en application						
US	OU5D(A&T) of/du 15.06.01	STANAG	06.01	06.01	06.01	06.01	06.01	06.01

* See reservations overleaf/voir réserves au verso

+See comments overleaf/Voir commentaires au verso

X-Service(s) implementing/Armées mettant en application

